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MANAGERS DESIGNERS/CONSULTANTS

**MEMORANDUM**

DATE: 2 December 1998

TO: David Bennett, WAM, U.S. EPA, Region X

FROM: Michelle Turner, Chemist, WESTON, Seattle
Roger McGinnis, Senior Environmental Chemist, WESTON, Seattle

SUBJECT: Validation of Organotin Data
Laboratory Batch: K9805617
Site: Duwamish River

WORK ASSIGNMENT NO: 46-35-0JZZ

WORK ORDER NO.: 4000-019-038-5200-00

DOC. CONTROL NO.: 4000-019-038-AAAK

cc. Bruce Woods, RAP-WAM, U.S. EPA, Region X
Dena Hughes, Site Manager, WESTON, Seattle (memo only)
Kevin Mundell-Jackson, Database Management, WESTON

The quality assurance review of four sediment samples, laboratory batch K9805617, collected from the Duwamish River has been completed. The sediment samples were analyzed for organotins by Columbia Analytical Services of Kelso, Washington. Samples were analyzed by gas chromatography with an FPD detector. The samples were numbered:

98344063 98344065 98344067 98344069

Data Qualifications

The following comments refer to the laboratory performance in meeting the quality control criteria described in the technical specifications of the laboratory subcontract. The review follows the format described in the *National Functional Guidelines for Organic Data Review* (EPA OSWER Directive 9240 1, February 1994), modified to include specific requirements of analytical methods

1. Timeliness

Holding time limits of 7 days for sample extraction and additional 7 days for analysis were established in the project Sampling and Analysis plan. All samples met holding time criteria.

2. Detection Limits

Instrument detection limits met project required quantitation limits.

3. Initial Calibration

A six-point initial calibration was performed prior to each analytical batch The percent relative standard deviation for the initial calibration was within limits of less than 25 percent RSD.

4. Continuing Calibrations

Continuing calibration check was performed after every 10 samples. All target analytes were within required limits for the continuing calibrations with the percent difference for a mid-range standard less than 25 percent.

5 Blanks

a) Laboratory Method Blanks

Laboratory method blank frequency criteria were met No target analytes were reported in laboratory method blanks.

b) Field Blanks

No field blanks were associated with this SDG.

6 Surrogate Compound Recovery

Surrogate recovery goals for tri-n-propyltin were established in the project Sampling and Analysis Plan at 60 to 130 percent for sediment. Based on conversations with the

laboratory an additional surrogate, tripentyltin was added and historical laboratory control chart limits were also used for data qualification. Laboratory limits are presented below:

| Surrogate Compound | Sediment Limits |
|---|---|
| Tripropyltin | 20 - 195% |
| Tripentyltin | 20 - 172% |

Surrogate compound percent recovery met quality control criteria for all samples, with the exception of the following:

| Sample | Surrogate | Percent Recovery |
|---|---|---|
| K9805624-006DMS (Batch QC) | Tri-n-propyltin | Not Calculated |

The Tri-n-propyltin surrogate recovery was not calculated due to matrix interference and high concentrations of analytes in the sample. As surrogate recoveries in the unspiked sample were within QC limits, no qualifiers were assigned.

7 Laboratory Control Sample (LCS)

LCS recovery goals for butyltins were established in the project Sampling and Analysis Plan at 60 to 130% for sediment. Based on conversations with the laboratory, historical control chart limits of 20 to 164 percent for sediment were also used for data qualification

Laboratory control sample percent recoveries met QC guidelines (P-project, L-laboratory), with the exception of the following:

| LCS | Analyte | Percent Recovery | QC Limit | Associated Samples |
|---|---|---|---|---|
| K980827-LCS | Di-n-butyltin | 40 | 60-130 (P)<br>20-164 (L) | 98344063<br>98344065<br>98344067<br>98344069 |
| K980827-LCS | n-Butyltin | 20 | 60-130 (P)<br>20-164 (L) | 98344063<br>98344065<br>98344067<br>98344069 |

Sample results were qualified as estimated (J) when LCS recoveries were outside project limits. Undetected results were qualified as estimated (UJ) when LCS recoveries were outside project limits.

8 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

The following matrix spike recovery goals were established in the project Sampling and Analysis Plan at for sediment.

| Analyte | % Recovery |
|---|---|
| Tributyltin | 40 - 120% |
| Dibutyltin | 30 - 120% |
| Monbutyltin | 10 - 129% |

MS/MSD sample percent recoveries for Tetra-n-butyltin met QC guidelines The relative percent difference (RPD) for Tetra-n-butyltin was 75 percent MS/MSD recoveries and RPDs were not calculated for Tri-n-butyltin, Di-n-butyltin and n-Butyltin as the analyte concentration was significantly higher than the spike level. No qualifiers were assigned solely on MS/MSD results.

9. Field Duplicate Analysis

No field duplicates were associated with this sample delivery group

10. Sample Analysis

A cursory review of raw data was performed. Deliverables were complete. A duplicate analysis was performed on Batch QC sample K9805693-001. RPDs between replicates were all greater than 35 percent. As this replicate sample was Batch QC, no qualifiers were assigned to samples based on laboratory replicate results. The case narrative indicates that the MS/DMS recoveries of mono-, di- and tributyltin for the Batch QC were not calculated as the analyte concentrations were significantly higher than the added spike solution. The high analyte levels prevented accurate evaluation of the spike recovery. Also, due to this high analyte concentration and matrix interference, surrogate recoveries for the DMS were not calculated. No other problems were noted.

11. Laboratory Contact

No laboratory contact was required.

Data Assessment

Upon consideration of the data qualifications noted above, the data are ACCEPTABLE for use except where flagged with data qualifiers that modify the usefulness of the individual values.

Data Qualifiers

U - The compound was analyzed for, but was not detected.

UJ - The compound was analyzed for, but was not detected The associated quantitation limit is an estimate because quality control criteria were not met.

J - The analyte was positively identified, but the associated numerical value is an estimated quantity because quality control criteria were not met or because concentrations reported are less than the quantitation limit or lowest calibration standard.

R - Quality control indicates that data are unusable (compound may or may not be present). Resampling and reanalysis are necessary for verification.

N - Presumptive evidence of presence of material (tentative identification).

I - Elevated reporting limit due to matrix interference.

**COLUMBIA ANALYTICAL SERVICES, INC.**

Analytical Report

| | | | |
|---|---|---|---|
| **Client:** | Roy F Weston, Inc | **Service Request:** | K9805617 |
| **Project:** | Duwamish River/4000-027-001-2019-38 | **Date Collected:** | 8/19/98 |
| **Sample Matrix:** | Sediment | **Date Received:** | 8/20/98 |

Butyltins

| | | | |
|---|---|---|---|
| Sample Name | 98344063 | Units | ug/Kg (ppb) |
| Lab Code | K9805617-007 | Basis | Dry |
| Test Notes | D | | |

| Analyte | Prep Method | Analysis Method | MRL | Dilution Factor | Date Extracted | Date Analyzed | Result | Result Notes |
|---|---|---|---|---|---|---|---|---|
| Tetra-n-butyltin | Method | Butyltins | 10 | 10 | 8/25/98 | 8/28/98 | ND | |
| Tri-n-butyltin | Method | Butyltins | 10 | 10 | 8/25/98 | 8/28/98 | 27 | |
| Di-n-butyltin | Method | Butyltins | 10 | 10 | 8/25/98 | 8/28/98 | ND 10 UJ | |
| n-Butyltin | Method | Butyltins | 10 | 10 | 8/25/98 | 8/28/98 | 11 J | |

D The MRL is elevated because of matrix interferences and because the sample required diluting

MQT 11/23/98

Approved By _____ Date 10/8/98

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## COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

| | | | |
|---|---|---|---|
| **Client:** | Roy F Weston, Inc | **Service Request:** | K9805617 |
| **Project:** | Duwamish River/4000-027-001-2019-38 | **Date Collected:** | 8/19/98 |
| **Sample Matrix:** | Sediment | **Date Received:** | 8/20/98 |

### Butyltins

| | | | |
|---|---|---|---|
| Sample Name | 98344065 | Units | ug/Kg (ppb) |
| Lab Code | K9805617-008 | Basis | Dry |
| Test Notes | D | | |

| Analyte | Prep Method | Analysis Method | MRL | Dilution Factor | Date Extracted | Date Analyzed | Result | Result Notes |
|---|---|---|---|---|---|---|---|---|
| Tetra-n-butyltin | Method | Butyltins | 10 | 10 | 8/25/98 | 8/29/98 | ND | |
| Tri-n-butyltin | Method | Butyltins | 10 | 10 | 8/25/98 | 8/29/98 | 33 | |
| Di-n-butyltin | Method | Butyltins | 10 | 10 | 8/25/98 | 8/29/98 | ND 10UJ | |
| n-Butyltin | Method | Butyltins | 10 | 10 | 8/25/98 | 8/29/98 | 14 J | |

D The MRL is elevated because of matrix interferences and because the sample required diluting

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**COLUMBIA ANALYTICAL SERVICES, INC.**

Analytical Report

| | | | |
|---|---|---|---|
| **Client:** | Roy F Weston, Inc | **Service Request:** | K9805617 |
| **Project:** | Duwamish River/4000-027-001-2019-38 | **Date Collected:** | 8/19/98 |
| **Sample Matrix:** | Sediment | **Date Received:** | 8/20/98 |

Butyltins

| | | | |
|---|---|---|---|
| Sample Name | 98344067 | Units | ug/Kg (ppb) |
| Lab Code | K9805617-011 | Basis | Dry |
| Test Notes | D | | |

| Analyte | Prep Method | Analysis Method | MRL | Dilution Factor | Date Extracted | Date Analyzed | Result | Result Notes |
|---|---|---|---|---|---|---|---|---|
| Tetra-n-butyltin | Method | Butyltins | 10 | 10 | 8/25/98 | 8/29/98 | ND | |
| Tri-n-butyltin | Method | Butyltins | 10 | 10 | 8/25/98 | 8/29/98 | 14 | |
| Di-n-butyltin | Method | Butyltins | 10 | 10 | 8/25/98 | 8/29/98 | ND | 10UJ |
| n-Butyltin | Method | Butyltins | 10 | 10 | 8/25/98 | 8/29/98 | ND | 10UJ |

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**COLUMBIA ANALYTICAL SERVICES, INC.**

Analytical Report

| | | | |
|---|---|---|---|
| **Client:** | Roy F Weston, Inc | **Service Request:** | K9805617 |
| **Project:** | Duwamish River/4000-027-001-2019-38 | **Date Collected:** | 8/19/98 |
| **Sample Matrix:** | Sediment | **Date Received:** | 8/20/98 |

Butyltins

| | | | |
|---|---|---|---|
| Sample Name | 98344069 | Units | ug/Kg (ppb) |
| Lab Code | K9805617-013 | Basis | Dry |
| Test Notes | D | | |

| Analyte | Prep Method | Analysis Method | MRL | Dilution Factor | Date Extracted | Date Analyzed | Result | Result Notes |
|---|---|---|---|---|---|---|---|---|
| Tetra-n-butyltin | Method | Butyltins | 10 | 10 | 8/25/98 | 8/29/98 | ND | |
| Tri-n-butyltin | Method | Butyltins | 10 | 10 | 8/25/98 | 8/29/98 | 37 | |
| Di-n-butyltin | Method | Butyltins | 10 | 10 | 8/25/98 | 8/29/98 | ND 10 U J | |
| n-Butyltin | Method | Butyltins | 10 | 10 | 8/25/98 | 8/29/98 | ND ↓ | |

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